Micro-beam XRF localization by a laser beam

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Abstract A new method for micro-beam XRF localization is presented. A laser beam along with an incident X-ray hits on the surface of a sample. The micro region on the sample that reached by X-ray beam can be localized by means of the visible spot of the laser beam. This method is suitable for X-ray microprobes using an X-ray tube or synchrotron radiation as excitation sources.
Keywords Localization for X-ray micro-beam, Laser beam

1 Introduction

Microprobes have been developed in order to analyze the chemical composition of samples in micro region. Within the past decade, X-ray microprobes were also evolved into a significant analytical tool, whereby excited fluorescent X-rays were monitored as a function of position in a sample. Several methods are used to obtain a microfocused X-ray source. Glass capillaries can be employed to focus X-ray from either a commercial anode or a synchrotron source for a higher X-ray flux[1~4]. The most ordinary way is to place an aperture in front of the source[5,6]. In addition to requiring minimal sample preparation and being nondestructive, microprobes based on the X-ray fluorescence (XRF) have also some advantages over conventional electron microprobes for acquiring element distribution from a sample[7], including higher sensitivity, greater penetrating depth and the ability of operation in air. X-ray microprobes have been used widely in many fields such as geology, biology, and microelectronics. Now X-ray microprobes has become one of the most powerful tools for microanalysis.

The spot of the micro-beam X-ray can not be seen. How can we localize the micro-beam spot of X-ray precisely on the surface of a sample? Several methods have been used to settle this problem. Nichol *et al.*[5], adopted beam monitor to survey micro-beam X-ray. Le *et al.*[8], moved the samples and a fluorescent glass in turn to determine the exact position of the X-ray spot. In these methods, two steps had to be adopted for micro-beam X-ray localization. First, the spot position of the micro-beam was determined, and then the samples were moved to the exact position of the beam. These methods are difficult to operate, and little changes of the position and the angle of the reflecting mirror or the microscopes can cause serious deviation of the localization.

A new system for micro-beam XRF localization has been developed in our laboratory, and the validity has been tested. A visible laser beam goes through the same collimator along with the X-ray beam. Both beams hit at the surface of a sample at the same point simultaneously. A visible spot of the laser beam pinpoints the micro-region that is bombarded by the X-ray beam. We can survey precisely the position of the X-ray micro-beam spot on the sample by visual inspection of the laser spot.

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2 Working principles

In Fig.1(a), a beam of laser 2 goes through the channel in the base of laser transmitter 4, and shoots at the reflecting mirror 9 tilted 45°. changing the incident direction by 90°. It then passes, along a perpendicular channel in the base, through the aperture of the collimator 6 and finally hits at the surface of samples 12. A laser spot can be seen on the sample. The operator can watch the position and size of the visible laser spot by a microscope at the direction 13 by means of mirror 10. A beam of X-ray 1 goes through the channel in the base of the X-ray tube and shoots at the mirror 9. We can let most of the X-ray transmit through the mirror by selecting suitable materials and thickness of the mirror. The X-ray beam passes through the aperture in the collimator and the hole in the reflecting mirror 10 after transmitting the mirror 9. It reaches the surface of the sample at the same point as the laser beam. We can localize X-ray beam spot on the sample surface by watching the visible spot of the laser.

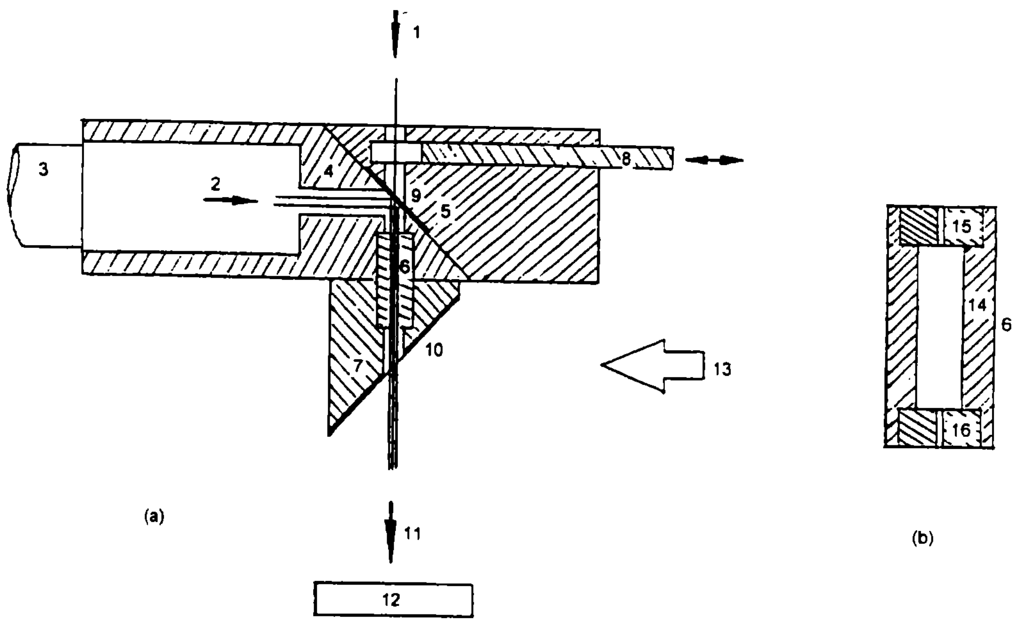


Fig.1 Schematic diagram of laser localization system
(a) Structure of the localization system (b) Structure of the collimator
1 Primary x-ray beam, 2 Laser beam, 3 Laser transmitter, 4 Base of laser transmitter, 5 X-ray channel, 6 Collimator, 7 Base of reflecting mirror, 8 X-ray shutter, 9 Reflecting mirror for laser beam, 10 Reflecting mirror of microscopes, 11 Line of x-ray and laser, 12 Sample, 13 Visual direction, 14 Collimator, 15 Upper collimator, 16 Down collimator

The mirror 9 plays double roles in the apparatus: (1) total reflecting laser beam; (2) little absorption of the primary X-ray. It should be able to endure radiation of high intensity of X-ray. In addition, it should have good property of conducting and dispersing heat produced by the X-ray. The materials of the mirror 9 should be carefully chosen.

A structure of double apertures was

adopted in order to improve the collimation of the X-ray micro-beam. It is demonstrated in diagram figure 1 (b). Apertures 15 and 16 determine the diameters of the laser and the X-ray beam. If the collimator were made of brass, the thickness of the apertures would be at least 3 centimeters. The distance between the apertures is twenty times longer than the diameter of the apertures. A strobe 8 is shut after completing each experiment so that the operator would not be damaged by the x-ray radiation.

The reflecting mirror 10 is made of ordinary material, brass, for example, in the system. We have used a very thin mirror with a pinhole in the middle for the laser and X-ray beam to traverse. This has no effect on the monitoring of laser spot by a microscope.

The inner diameter of channel in the base of the laser tube is bigger than the outer diameter of the laser tube. The laser tube is hold by eight screws. By adjusting these screws we can control the direction and position of the laser beam and align it with the X-ray beam after reflecting by the mirror 9.

3 Experimental results

3.1 The validity of the localization system

By means of a microscope, the location of the laser spot on the surface of the sample stage can be watched clearly. A schematic diagram of the visual field is demonstrated in Fig.2. The diameter of the aperture in the collimator is 100 μm. The micro region covered by the laser spot is illustrated in Fig.2. By visual inspection the laser spot diameter is about 110μm. To survey the validity of the localization system, an experiment was conducted. Firstly we place a thread of brass (a) with a diameter of 25μm on the sample stage (Fig.2). The sample stage was moved horizontally with a step of 25μm to let the thread of brass (a) traverse the micro region covered by the laser spot. Because the primary X-ray bream goes along with the laser beam, the characteristic X-ray of Cu will be excited by it. For each step the thread of brass was measured for 60 seconds. The micro XRF spectra were acquired by a multichannel analyzer system based on a personal computer. Calculating the fluorescent counts of Cu K, we can get a distribution curve of the fluorescence counts in horizontal direction (Fig.3 left). Secondly we moved the sample stage perpendicularly to let the thread of brass (b) traverse the micro region covered by the laser spot. Repeat the above experiment and get another distribution curve (Fig. 3right). We have defined the FWHM as the spatial resolution of micro X-ray beam[8]. Fig.3a and Fig.3b show that the spatial resolution of the instrument is 85μm in perpendicular direction and 97μm in horizontal direction. Table 1 shows the coordinates of the micro-region covered by the laser and the X-ray

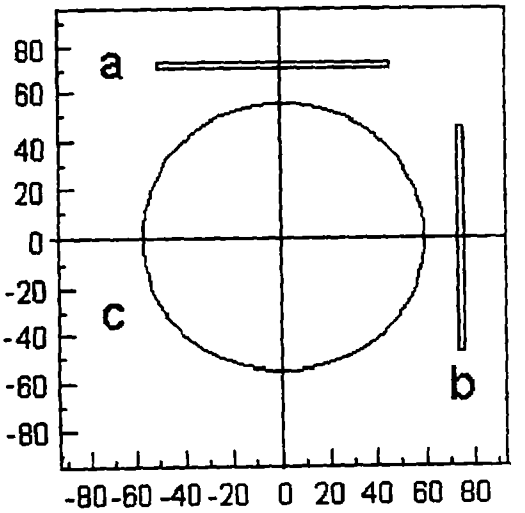
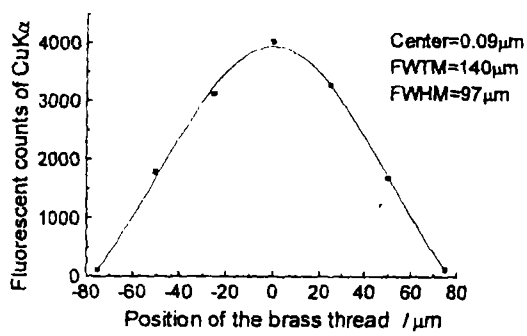


Fig.2 Visual field of the microscope
a,b A thread of brass,
c The laser spot

spot. According to the result of the experiment, the laser spot also covers the region that is covered by the X-ray spot. Although the size of the laser spot is smaller than that of the X-ray spot, the laser spot can point out the location of the X-ray spot. So we can localize the X-ray microbeam by the visual laser beam conveniently and correctly.



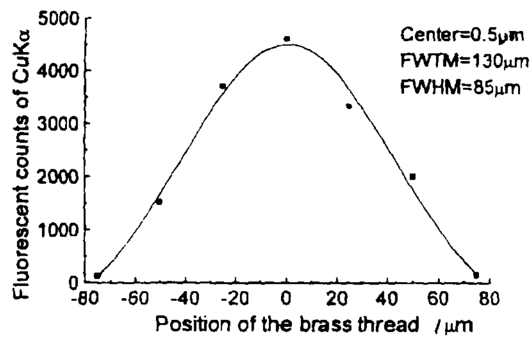


Fig.3 Size of the micro X-ray spot (left) Horizontal direction (right) Perpendicular direction

Table 1 Micro region of the laser and X-ray spot

	Coordinate/μm		Size of spot/μm
	x	y	
Laser spot	-55, 55	-55, 55	110×110
X-ray spot	-65, 65	-70, 70	130×140

3.2 Analysis of the manganese nodules in micro region

The concentration distribution of metal elements in various depths manganese nodules from deep ocean has been observed by means of micro-PIXE.[9] The element concentration maps in micrometer scale areas confirmed the earlier discovery that there exists strong coherence between Ni, Cu and Mn, and strong complementarity between Fe and Mn contents. In this experiment by means of the X-ray microprobe we try of find the relationship between these element in scanning spot areas of a little larger scale. Fig.4 shows the photomicrograph of a sample. The nodule sample was attached to a stage that could be moved in three dimensions. An optical microscope was positioned over the specimen stage, which is used to watch the longitudinal section of a nodule sample. With the help of the laser localization system we located the interested area easily and accurately. A total of twelve single spots were analyzed in various depths, representing different sedimentary periats, of manganese nodules. These spots were marked on Fig.4. Each spot is about 100 μm in size. The element concentration for Mn, Ni, Cu and Fe is illustrated in Fig.5. We could find the same phenomenon. Compared with micro-PIXE, X-ray microprobe with the laser localization system in more convenient to use, it can be used for intermediate-scale (scanning spot area) compositional mapping to bridge the gap in spatial resolution between bulk X-ray fluorescence and nuclear microprobe (or electronic microprobe) methods

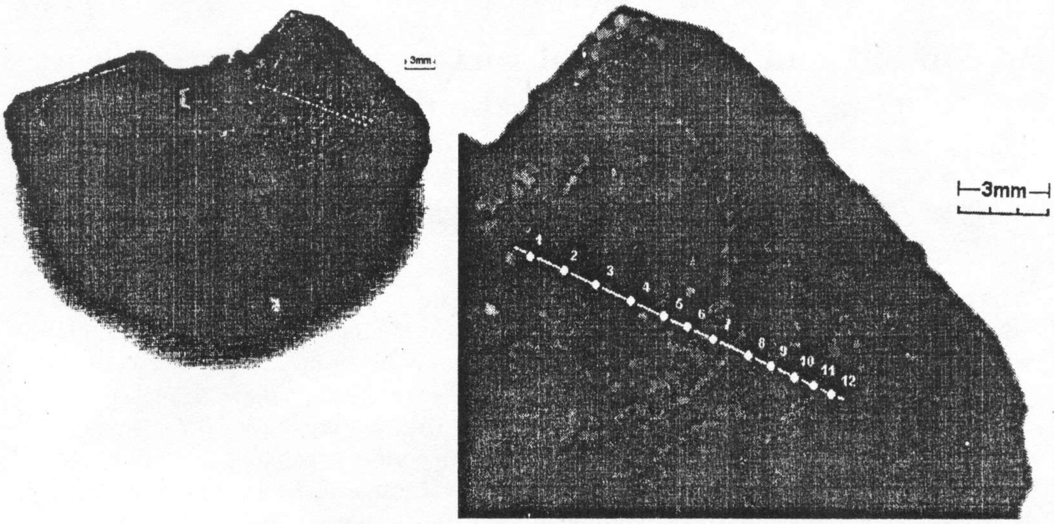


Fig.4 (Left) The photomicrograph of the longitudinal section of a manganese nodule sample showing the epoch rings of different sedimentary periods (Right) A part in its enlarged image, wherein the labels (1, 2 ⋯ 12) are the marks of measurement points

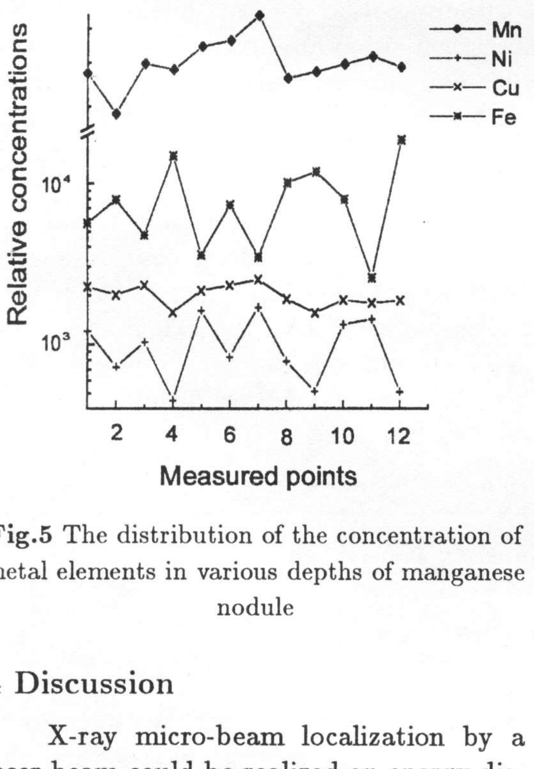


Fig.5 The distribution of the concentration of metal elements in various depths of manganese nodule

4 Discussion

X-ray micro-beam localization by a laser beam could be realized on energy dispersive fluorescence spectrometer using X-ray tube or synchrotron radiation as excitation sources. It helps the operator to locate an interested region directly. Compared with other method of X-ray localization, the laser localization system presented here is more convenient and accurate.

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